



# National Institute of Standards & Technology

## Certificate of Analysis

### Standard Reference Material 136e

#### Potassium Dichromate



#### Oxidimetric Standard

This Standard Reference Material (SRM) consists of high-purity potassium dichromate and is intended primarily for use in oxidimetric standardization. It conforms to the American Chemical Society specifications for analytical reagent grade material, and meets the primary standard criteria of the Analytical Chemistry Section of the International Union of Pure and Applied Chemistry [Analyst 90, 251 (1965)].

The certification of this lot of potassium dichromate was a cooperative effort of the National Institute of Standard and Technology (NIST) and the U.S. Department of Energy New Brunswick Laboratory (NBL), Argonne, Illinois.

The certified value is the weighted mean of results from three different analytical methods (see Analysis). The weights were computed according to the iterative procedure described in Paule and Mandel (NBS Journal of Research 87, 1982, pp. 377-385).

#### Certified Value

Oxidimetric Assay - - - - - 99.984  $\pm$  0.010 weight percent

The uncertainty is the half-width of an approximate 95% confidence interval for the certified value plus an allowance for bias among the analytical techniques.

The molecular weight of  $\text{K}_2\text{Cr}_2\text{O}_7$  used in all calculations was 294.1844 and the density used in computing its mass in vacuum was 2.69  $\text{g}\cdot\text{cm}^{-3}$ .

The experimental sequence was developed by S.B. Schiller of the Statistical Engineering Division, who also statistically evaluated the results. Analytical measurements were performed by George Marinenko of the Inorganic Analytical Research Division.

The cooperative measurement effort at NBL was coordinated by N.M. Trahey, Manager, NBL Reference Materials Program. The measurement plan for titrimetric assay was developed by P.M. Santoliquido, Safeguards Instrumental Measurement Division; the high-precision version of NBL-modified titrimetric method for uranium was performed by W. Nichiporuk, Safeguards Measurement and Development Division; and the statistical scheme and calculations were generated by M.M. Smith, Operations Support Division.

The technical and support aspects involved in the procurement, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R.W. Seward.

June 1, 1989  
Gaithersburg, MD 20899

Stanley D. Rasberry, Chief  
Office of Standard Reference Materials

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## ANALYSIS

Coulometric Assay: The coulometric assay of SRM 136e is based on the reduction of the dichromate ion in 500 mg samples. The coulometric procedure used in this analysis has been described by G. Marinenko and J.K. Taylor, High Precision Titration of Potassium Dichromate, J. Res. Nat. Bur. Stand. (U.S.), 67A(5), 453-459, 1963 (September-October). The value of the Faraday constant used in the calculation was  $96486.0 \text{ C}_{\text{NIST}} \text{ mol}^{-1}$ .

Titrimetric Assay: SRM 136e was analyzed at NBL using the high-precision version of the NBL-modified titrimetric method for uranium. NBL CRM 112-A (formerly SRM 960), Uranium Metal Standard, was used in the analysis of ten bottles of 136e in duplicate or triplicate along with samples of CRM 99 (NBL Potassium Dichromate Standard) and SRM 136c (a previous lot of the Potassium Dichromate Standard), which were used for controls.

Gravimetric Titration Assay: Six of the same bottles of 136e used for the coulometric assay were intercompared with SRM 136c by a weight titration with ferrous ammonium sulfate. While this method is not as precise as either the coulometric method or the NBL-modified titrimetric method, it does provide an independent assay method.

Homogeneity: This SRM lot of potassium dichromate is homogeneous within the bounds of the random error uncertainty of the measurement process.

Drying: This material may be used as received or after drying. However, it is recommended that this material be dried at  $110^\circ\text{C}$  for 2 hours.

Stability: SRM 136e is stable and the certification is valid for five years from the date of shipment, when stored in its original container with the top tightly closed. Should any change occur before the expiration of the certification, NIST will notify purchasers.

This lot of potassium dichromate was obtained from Mallinckrodt, Inc., St. Louis, Missouri.